STUDIES IN THE ANALYTICAL CHEMISTRY OF SODIUM

II. RESORCINOL SULPHONIC ACID AS A REAGENT FOR SODIUM

by

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Resorcinol sulphonic acid has been recommended by BARRETO[1] as a qualitative and quantitative reagent for sodium. The original paper was not available in Britain and details of the method could only be obtained from an abstract.

BARRETO recommends that tests be made by adding to a 20% solution of the salt to be tested, 25 ml of the reagent for each gram of salt present. The precipitate is allowed to stand for 30 min. and filtered and washed successively with the reagent, ethanol and ether. It is then dried and weighed. Although potassium produces a similar precipitate, it is readily soluble in excess of the reagent.

It appears from the foregoing that a fairly concentrated test solution must be used. This may not be convenient in practice, especially if small amounts of sodium are to be subjected to the test. For example, if 8 mg of sodium were present—an amount readily detected or determined by the zinc uranyl acetate method—in the form of sodium chloride, it would be necessary to dissolve the salt in 0.1 ml of water to obtain approximately the stipulated 20% solution. The amount of reagent to be added to this amount of sodium chloride would be 0.5 ml and thus it would be necessary to apply micro-filtration methods for a quantity of sodium which can be filtered by the normal procedure when the conventional reagents are used. There is no indication in the abstract which ions interfere other than potassium.

We have examined the reagent under the more usual conditions of precipitation, but it is not very sensitive. The minimum amount of sodium which could be detected was 10 mg per ml of test solution. This represents roughly a 2.5% solution of sodium chloride as distinct from the 20% solution stipulated by BARRETO. On the other hand, a considerable excess of reagent over and above that stipulated for the amount of salt present, had to be added to obtain a positive reaction. It is also possible that precipitation is not complete under these conditions. Presumably if concentrations less than 10 mg per ml have to be detected, reduction of the volume of the test solution would enable a positive test to be obtained.

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However, a solution containing 1 mg of sodium would have to be reduced to 0.1 ml and it would not be easy to see a faint white precipitate in such a volume.

Potassium, magnesium, and lithium did not interfere with the test, but because of the poor sensitivity the reagent cannot be recommended for the detection of sodium, although it works satisfactorily under the conditions proposed by BARRETO. It is possible that some improvement might result by the use of a purer reagent. With BARRETO's method of preparation some unchanged resorcinol and free sulphuric acid will probably be present and may have an inhibitory action on the test.

EXPERIMENTAL

The reagent was prepared by the method described by BARRETO.

100 g of resorcinol were heated with 500 g of concentrated sulphuric acid at 120° C during 1 hour. The mixture was then cooled to 30° C, 500 g of crushed ice added and stirred until all the ice melted.

Tests were carried out by adding various amounts of the reagent to 2 ml portions of the test solution which contained varying amounts of sodium ion.

The results are included in Table I.

TABLE I

THE REACTION OF RESORCINOL SULPHONIC ACID WITH SODIUM, POTASSIUM, MAGNESIUM, AND LITHIUM CHLORIDES

Test soln ml	Na^+ present mg	K^+ present mg	Li^+ present mg	Mg^{+2} present mg	Reagent ml	Result
2	—	160	—	—	2	No precipitate
2	—	—	160	—	2	id.
2	—	—	—	160	2	id.
2	160	—	—	—	2	Immediate white precipitate
2	40	—	—	—	2	After 1 min vigorous shaking slight precipitate. Increased on standing
2	40	—	—	—	4	After 1 min vigorous shaking appreciable precipitate formed
2	20	—	—	—	2	No precipitate after standing 20 min
2	20	—	—	—	4	id.
2	20	—	—	—	6	id.
2	20	—	—	—	8	Turbidity after 5–10 min increased on standing to appreciable precipitate
2	16	—	—	—	8	No precipitate after standing 20 min
2	16	—	—	—	10	id.
2	16	—	—	—	12	id.
5	16	—	—	—	16	id.

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SUMMARY

Resorcinol sulphonic acid has been examined as a reagent for the detection of sodium. The minimum concentration at which a positive test could obtained was 10 mg of sodium per ml. Potassium, lithium, and magnesium ions do not precipitate under the conditions of the test, but the reaction is not sufficiently sensitive to be recommended as a test for sodium.

RÉSUMÉ

L'acide résorcinol sulfonique a été étudié en vue de son utilisation comme réactif du sodium. On peut déceler jusqu'à 10 mg de sodium par ml. Les ions du potassium, du lithium et du magnésium ne précipitent pas dans les conditions de l'essai, mais la réaction n'est pas assez sensible pour être recommandée pour la recherche du sodium.

ZUSAMMENFASSUNG

Resorcinsulfonsäure wurde als Reagens auf Natrium untersucht. Es können bis zu 10 mg Natrium pro ml nachgewiesen werden. Kalium- Lithium- und Magnesiumionen werden under den beschriebenen Versuchsbedingungen nicht gefällt, aber die Reaktion ist nicht genugend empfindlich um zum Nachweis von Natrium empfohlen werden zu können.

REFERENCES

[1] A. BARRETO, *Rev. chim. ind.* (*Rio de Janeiro*), 5 (1936) 416.

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